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## NBS Special Foreign Currency Program in Yugoslavia 1971-72

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# Special Foreign Currency Program in Yugoslavia 1971-72

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U.S. DEPARTMENT OF COMMERCE, Peter G. Peterson, Secretary  
NATIONAL BUREAU OF STANDARDS, Lawrence M. Kushner, Acting Director,

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## FOREWORD

This Publication reviews the National Bureau of Standards Special Foreign Currency Program in Yugoslavia. Its purpose is to allow interested scientists and scientific administrators to form opinions on the value of such programs from a number of points of view - from the standpoint of officials in the grantor and grantee countries and from that of a more general reader who is interested in examining an example of international cooperation and its success in contributing to the general betterment of the two countries involved and in improving international scientific relationships.

A summary of each of the projects funded under this Program is included in this report. The summaries list the project goals, some major results, the publications which resulted from the specific support under this Program and the names of the NBS monitor and the principal investigators. From the NBS viewpoint the Program is judged to have been scientifically productive and to have contributed to our mission objectives. We hope that the Yugoslav officials similarly conclude that our collaborative program has contributed to their own goals as well.

Further, we consider that opportunities abound for a long continuing program of mutual interest, and the report describes some of the specific projects that might be undertaken in the future. NBS intends to continue to explore these opportunities and we trust that the good technical associations that have developed under this program will continue and be strengthened in the future.

Lawrence M. Kushner  
Acting Director, National Bureau of Standards

## ABSTRACT

An overview is given of grants awarded by the National Bureau of Standards under the Special Foreign Currency Program (SFCP) in Yugoslavia, authorized by Public Law 480 and other legislation. Each grant is identified by title, principal investigator, institution in Yugoslavia, NBS monitor, and the monitor's organizational unit within NBS. The work is then described briefly under the three headings "Summary Description of Project Goals," "Results and Implications to Date" and "List of Publications that Resulted from the Project." To demonstrate the relevance of such grants to the programs of NBS, the grant descriptions are ordered by the elements of that Program Structure. The significance and purpose of the NBS/SFCP grant program are discussed in the Foreword, the Introduction and an Appendix. The NBS monitors and the program manager judge this grant program to have had a high benefit to cost ratio from the viewpoint of NBS.

Key words: Binational research cooperation; international scientific cooperation; physical science research administration; research planning; scientific research abstracts; Special Foreign Currency Program; Yugoslavia science and technology.

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## 1. INTRODUCTION

This Technical Note is intended to provide a review of the scientific and technological grants program of the National Bureau of Standards (NBS) conducted in Yugoslavia by use of the "Excess" Special Foreign Currency (SFCP) funds. Such information is needed for any value judgment, be it from the viewpoint of the disciplines, the institutions or the technological plans of the countries. Each grant currently funded by the NBS/SFCP is described briefly in terms of its initial project goals and its significant accomplishments and implications for science or technology, and especially for the aims of the cooperating institutions of both countries.

Additional projects presenting appropriate cooperative opportunities have been identified by Yugoslav scientists and engineers. Two examples, chosen at random, of such future attractive projects are described in Appendix I, in which the outlines of each project are given.

This Report is a direct outgrowth of the visit to Yugoslavia of four NBS staff members in October 1972. The team was under the leadership of Mr. H. S. Peiser, Chief of the NBS Office of International Relations and Manager of the NBS/SFC Program. Other team members were Dr. S. E. Chappell, Programs Coordinator in the Office of the Associate Director for Information Programs; Dr. E. Horowitz, Deputy Director, Institute for Materials Research, and Dr. H. Yakowitz, Metallurgist. The purposes of the visit were to promote further scientific and technological contacts; to seek mutually desirable and carefully selected exchanges of information and professional staff; to review and evaluate the SFCP supported projects; and to identify new proposals of excellence in the event funds become available for their support.

The NBS Special Foreign Currency Program discussed in this report can be traced to the initial visit to Yugoslavia of a five-man NBS scientific team in September (11-26), 1970. This team was under the leadership of Dr. E. Horowitz, Deputy Director of the NBS Institute of Materials Research. Other members of the team were Dr. H.S. Boyne, Chief, Quantum Electronics Division; Dr. E. G. Fuller, Chief, Photonuclear Physics Section; Mr. F. L. Hermach, then Chief, Electrical Instruments Section; and Dr. D. R. Lide, Chief,

Office of Standard Reference Data. This team accumulated firsthand information about science and technology in Yugoslavia based on discussions with scientists, engineers, educators, R&D administrators, and government officials. Visits to more than two dozen selected laboratories and institutes in eight cities including Belgrade, Zagreb, Ljubljana, Sarajevo, and Zenica were completed in the two week period. Recommendations were made to the Director of the National Bureau of Standards for funding projects in Yugoslavia under the Special Foreign Currency Program based on the team's evaluation of the project proposals in accordance with established criteria, and the capabilities of those submitting the proposals to undertake and successfully complete the work. The acceptance of these recommendations led directly to the establishment of the NBS-Yugoslav cooperative science and technology program.

The reader examining the Program from the grantor country's point of view has the opportunity to judge whether it has contributed to the goals of NBS appreciably and whether US national goals were served. To make this task easier we have assembled the sections of this Technical Note into categories identified by the NBS Program Structure under which all NBS projects are classified. The list of these Program Structures appears in Appendix II.

The current program is of direct benefit to at least five major elements of the NBS mission. Several of the projects are involved with measurement techniques not yet exploited at NBS. Input for the National Standard Reference Data System is provided in areas not yet being investigated at NBS. Many NBS projects are currently involved with computer control of experiments. Closely related is the project in Yugoslavia on interactive control between experiments and computer. Finally, some of the Yugoslav projects are concerned with the development of standards and standard reference materials, supplementing NBS work and providing both new and better characterized standards.

The Yugoslav oriented reader, on the other hand, should ask whether new and increased capabilities have been brought to Yugoslavia by these projects and whether the collaboration has been useful to institutional and national goals. Questions should be asked: Have new competences been built up and existing ones strengthened? Have scientific and technological talents been diverted from the national goals of Yugoslavia? Have neighboring fields been enriched in Yugoslavia by cross-fertilization? Have applications in industry been sparked

and has the Program helped to develop a broadly based infrastructure of science and technology in Yugoslavia?

Readers who are experts in the fields of investigation may ask such questions as:

1. Were the projects in the fields familiar to you well executed?
2. Did the senior investigators demonstrate exceptional competence which could be of special service in the future?
3. Did the projects contribute to the solution of problems of known importance to science, technology, and society?
4. Is there evidence that contact between US and Yugoslav scientists was intellectually stimulating and helpful in related work?
5. Does the program appear to have contributed to a stimulating climate for scientific progress and technological innovation?
6. Did the program help to widen the horizons of scientists towards application of their expertise to urgent national needs?
7. Does this report suggest that bi-national programs of this type should be developed for mutual benefit?

This list of questions is certainly not exhaustive nor are their answers by any means obvious.

This report is addressed most particularly to scientifically trained science administrators. In it the editors have summarized fourteen projects, supported by approximately \$200,000 in special foreign currency funds, plus matching funds of about the same magnitude provided by the Yugoslav institutions.

Several management details may be responsible for much of the success of this program. First of all, the NBS monitor has been strongly encouraged to communicate directly with the principal investigator. Secondly, proper scheduling of visits by the NBS monitors to Yugoslavia has broadened the basis of interaction. Thus, the input to the NBS/SFCP from the NBS monitor can be based upon his knowledge of the general Yugoslav scientific context of the particular project which he is monitoring. To promote widespread knowledge at NBS of Yugoslavian scientists and institutions, whenever it becomes necessary, for scientific reasons, for an NBS monitor to visit his project in Yugoslavia, he is urged to visit as many additional scientific institutions and



laboratories as possible. Such visits are to be described in writing, on his return, and include his view of the scientific competences and the particular strengths or weaknesses of individuals, laboratories, and institutions that he observed. In addition to serving to broaden the outlook of the particular NBS monitor, it increases the extent of the contacts between Yugoslav scientists and the NBS staff, thereby enabling the Yugoslav scientist to see NBS from the various points of view brought by the NBS visitors. This increases the likelihood that Yugoslav scientists involved in the program will, to a certain extent, insert an NBS point of view in addition to their own, into goals set for their NBS projects. By careful planning of such NBS staff visits their cost has been kept to less than 2 percent of the project funds available. The trip reports by the two NBS teams that visited Yugoslavia are recommended reading for NBS staff visiting Yugoslavia in the future.

The files of the program contain a growing number of examples of collaborative work which has been of direct utility to either or both sides. Such collaborative efforts are extremely important in today's world. The unit for scientific research is seldom an individual scientist, but rather a team. The complex and interdisciplinary nature of modern scientific work makes the use of self-contained, local expertise of utility a component in collaborative efforts which cross lines normally separating institutions, countries and areas of research.

However true these and other benefits from NBS collaboration with Yugoslav institutions may have been, the program as a whole must be judged on the scientific and technical merits of the individual projects. For that reason we have given more technical details than are normally submitted to science administrators. Review of this program is timely because the SFCP in Yugoslavia is at a critical state and crucial decisions about its continuation may be forthcoming in the near future. Consideration for other mechanisms for continued cooperation between the NBS staff and Yugoslav scientists may now also be in order.

The editors would greatly appreciate receiving any comments that the readers would care to make regarding any of the individual projects or the overall program.

A.1.\* PHYSICAL MEASUREMENT METHODS, UNITS AND STANDARDS

Project Title: FIRST INTERNATIONAL SUMMER SCHOOL ON RADIONUCLIDES METROLOGY

Principal Investigator: Dr. Dj. N. Bek-Uzarov

Institution: Boris Kidrič<sup>Y</sup> Institute, Vinca

NBS Monitor: Dr. W. B. Mann

NBS Institute or Center and Division: Institute for Basic Standards  
Center for Radiation Research

Summary Description of Project Goals

The goal was to carry out a Summer School which will bring together specialists in the field of Nuclear Metrology and adjacent disciplines in order to review and intensively discuss the relevant state of the art and possible future developments. The forum is to permit a wide exchange of information among workers in this field. The Proceedings of this first International Summer School on Radionuclides Metrology are to be published. The significance of the Summer School is expected to be expressed in the work towards an urgently needed international acceptance of the best available measurement techniques and to provide clear statements of their relevant reliabilities.

Results and Implications to Date

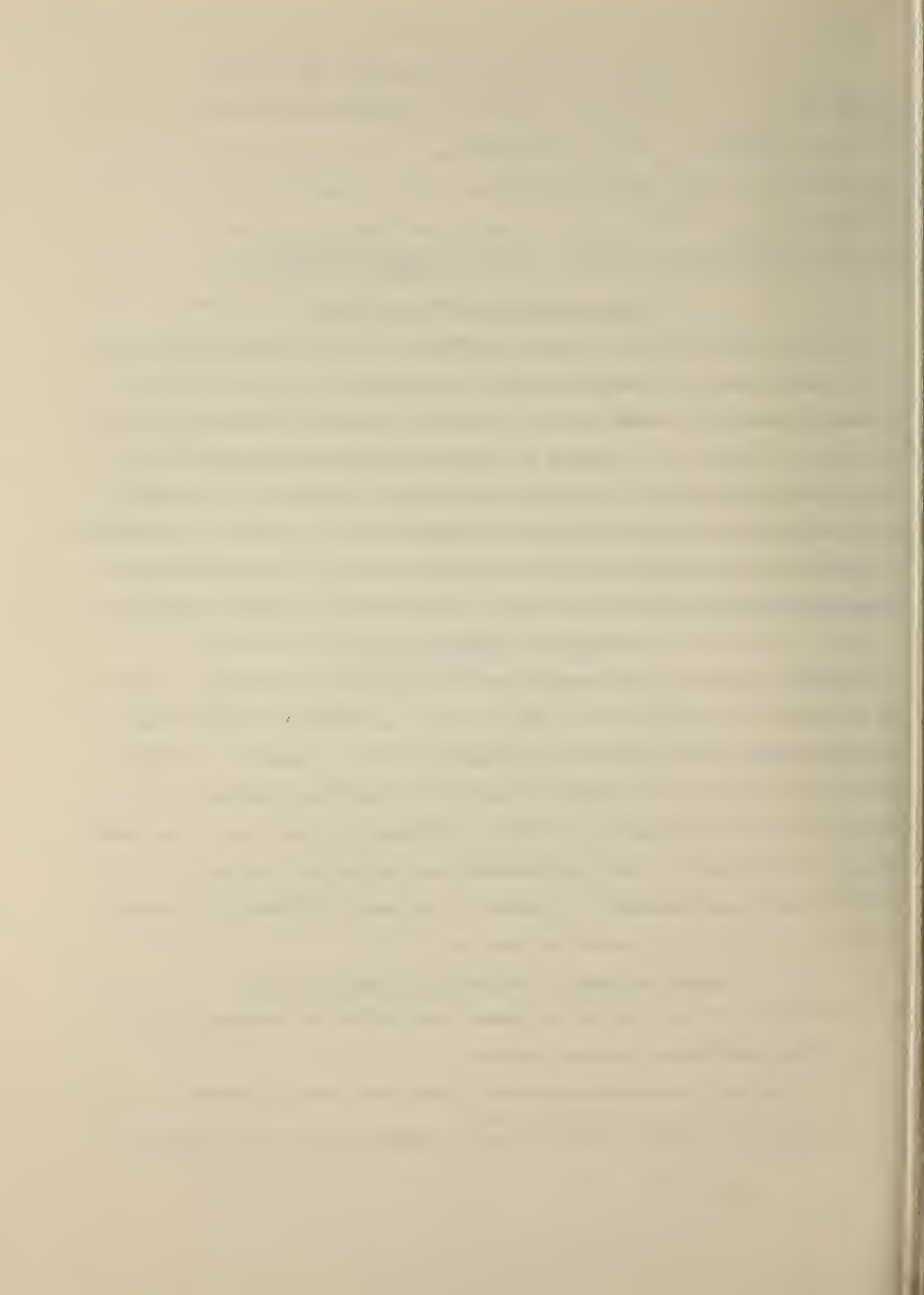
The Summer School was held at Herceg Novi from August 21 to September 1, 1972. It was attended by 105 experts from 23 countries as well as interested advanced students from all principal relevant laboratories in Yugoslavia. The consensus was that the school program achieved its objectives and contributed toward the wider acceptance of reliable methods of measurement in this field. The very approximate cost of the Summer School was \$25,000, of which this grant provided about 20 percent. The Boris Kidrič<sup>Y</sup> Institute itself contributed about 40 percent of the funds. The remaining 40 percent were provided by many other national laboratories.

List of Publications that Resulted from the Project

1. Proceedings of the 1st International Summer School on Radionuclides Metrology, J. Nuclear Instruments & Methods, in press.
2. Abstract Booklet of 51 lecture papers and 12 contributed research papers.

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\* The alphanumeric designation refers to the NBS Program Structure (see Appendix II).



Project Title: DETERMINATION OF IMPURITY LEVELS IN ANALYTICAL GRADE AND ULTRAPURE  
CHEMICALS BY ACTIVATION ANALYSIS USING TRIGA REACTOR

Principal Investigator: Professor Lado Kosta

Institution: Jožef Stefan Institute, Ljubljana

NBS Monitor: Dr. W. Wayne Meinke

NBS Institute or Center and Division: Institute for Materials Research  
Analytical Chemistry Division

#### Summary Description of Project Goals

The project originally involved the characterization of European produced chemical reagents of high purity for a number of trace elements. The emphasis has now shifted by mutual arrangement to (1) the analysis of Standard Reference Materials, or materials intended as such, and in particular to the NBS range of Biological Reference Materials and the trace elements in Glass SRM's and (2) the development of analytical methods for such materials. The principal method of investigation will be neutron activation analysis which will be supplemented by atomic absorption, anodic stripping polarography and spectrophotometry.

#### Results and Implications to Date

Methods have been developed for checking the trace elements in some chemical reagents, such as mineral acids, ammonia and hydrogen peroxide; and for these the presence of As, Cl, Co, Cr, Cu, Fe, Mn, Na and Zn has been quantitatively determined. Irradiations of medium to long exposure are required for the elements producing radionuclides of longer half-life. In those instances radiation decomposition with consequent pressure build-up in the ampoules constitutes a hazard. To prevent this, the samples are evaporated prior to irradiation. A special closed evaporator, working under chemically isolated conditions was built for this purpose.

For biological materials, namely orchard leaves, bovine liver, tomato leaves and coal, emphasis in the neutron activation work has been placed on important trace elements which give rise to nuclides of short or medium half-life, in particular

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\* The alphanumeric designation refers to the NBS Program Structure (see Appendix II).



As, Cu, Hg, Mn, Sb, Sn, V and Zn (as  $^{69}\text{Zn}$ ). For Hg, a volatilization technique developed in this laboratory has been used. A new method for V has been worked out, which avoids the frequently used pre-irradiation dry-ashing step, with its associated uncertainties arising from appreciable volatility of some vanadium compounds. For As, Sb and Sn, specific post-irradiation solvent extraction separations are used based on an investigation of the  $\text{H}_2\text{SO}_4$ -KI/ toluene system. Cu, Mn and Zn determinations are also based on selective solvent extraction procedures. All separated radionuclides are counted in a NaI(Tl) well-type, scintillation detector and a multichannel analyzer system which yields the highest attainable sensitivity and minimizes geometrical effects.

This non-destructive approach to trace analysis should yield information on elements such as Na, K, Mn, Br, Cl, and Ca. In the case of coal, As, V, La and some rare earth elements such as Eu and Sm can be so determined. In some other specimens Co, Cr, Cs, Fe, Rb, Sc, Se and Zn are being analyzed after long, high-dose irradiations. As mentioned below under glasses, the accuracy of the information obtained from such measurements is probably lower than from radio-chemical determinations. The accuracy of the multi-element approach will be increased in the near future by the acquisition of a larger multi-channel analyzer which has data-reduction capability.

Atomic absorption in the visible and uv region is used as a cross check to determine the quantity of Na, K, Mg; Ca, Cd, Cu, Fe, Mn, and Zn in the biological materials. All these elements, except Cd, are found in concentrations above 10 ppm in most of these samples. Atomic absorption is also used for determining yields in some radiochemical processes.

Anodic stripping polarography applied directly to the solution obtained from wet ashing of biological material has been developed as another cross-check for determining trace quantities of Cd, Cu, Pb and Zn, while conventional spectrophotometric methods are also used for Cu and Fe.

Of the glass SRM's to be supplied by NBS, the two lowest ranges of trace element contents of nominally 0.02 and 1 ppm are being investigated. Because of the high

matrix activity from  $^{24}\text{Na}$  and the difficulties associated with its elimination, combined with the complex radioactivity mixture resulting from the 60 added elements, the problems associated with the analysis are severe in these two ranges. In these cases, emphasis is being given to developing methods mainly by destructive radiochemical techniques and obtaining results for elements that have not yet been certified by the NBS. Nondestructive techniques are of limited value, but may be applicable to long-lived nuclides of Co, Cs, Ir, Rb, Sc, Ta and some rare earths such as Eu and Tb. With the relatively low counting efficiency of Ge(Li) detectors and the limited sensitivity for elements such as Co, Cr, Fe and Zn at available fluxes, nearly all the work so far has been concerned with the destructive approach. In view of the uncertainties associated with nondestructive techniques, such as poor statistics, geometrical and self-absorption problems, and pulse-pile-up from the dominant activities, the radiochemical approach is probably preferable from the point of view of accuracy, especially for the 0.02 ppm range.

To achieve sufficient induced activity, the 0.02 and 1.0 ppm level glasses are irradiated for the long-lived radionuclides in the reactor at Vinca, Belgrade, at a flux of  $1.6 \times 10^{13} \text{ n cm}^{-2} \text{ sec}^{-1}$  for about 200 hours. For shorter-lived radionuclides such as  $^{64}\text{Cu}$ ,  $^{56}\text{Mn}$ ,  $^{76}\text{As}$ ,  $^{198}\text{Au}$ , the Institute Jôzef Stefan TRIGA Mark II is used for periods from 2- 20 hours at 250 kW. After dissolution of the irradiated glasses in HF/mineral acid mixtures,  $^{24}\text{Na}$  is removed by passage through a column of hydrated antimony pentoxide (HAP). For the shorter-lived radionuclides procedures similar to those used for biological materials are employed. With long-lived Co, Cr, Fe, and Zn, an ion exchange separation forms the main stage of the analysis.

For elements, like As, for which losses due to volatility would otherwise occur the matrix must be dissolved in a closed system. These results are of interest since they should show whether volatile elements are uniformly distributed in all the glass batches, or whether losses occur in the extended heating of the melt during the rod drawing process used in the preparation of the wafers.

In summary, the quantitative analysis of many trace elements has been achieved on chemical reagents of high purity, on biological specimens, and on standard

reference materials consisting of glasses with incorporated trace quantities of metal ions. The even wider use of trace analysis in the characterization of materials and the increased recognition that the properties of materials are often dependent upon the presence of trace elements, makes this project very timely. Thus, more and more, the quality control of certain materials important to industry and science depends on such basic work in trace analysis for the certification of accurately measured standard reference materials.

List of Publications that Resulted from the Project

1. L. Kosta and V. Ravnik, "Application of Activation Analysis for the Determination of Cl, As, Cu, Mn, Na in Mineral Acids, Ammonia and Hydrogen Peroxide," *Radioanal. Letters* 7, 295 (1971).
2. L. Kosta and V. Ravnik, "Determination of Trace Quantities of Iron, Chromium, Cobalt and Zinc in Very Pure Ammonia, Hydrogen Peroxide and Mineral Acids by Neutron Activation Analysis," to be published in *Vestnik Slov. Kem. Drustva* (1972).
3. M. Levstek, L. Kosta, M. Dermelj and A. R. Byrne, "Vanadium Determination in Biological Materials by the use of Preconcentration," Paper SM-157/39, Proceedings of 2nd IAEA Symposium on Nuclear Activation Techniques in the Life Sciences, Bled (April 1972).
4. A. R. Byrne and D. Gorenc, "The Toluene Extraction of Some Elements as Iodides from Sulphuric Acid-Potassium Iodide Media. Application to Neutron Activation Analysis," *Anal. Chim. Acta* 59, 91 (1972).
5. A. R. Byrne, "idem, Part II. Determination of As and Sb in Biological Materials at Submicrogram Levels," *Anal. Chim. Acta* 59, 91 (1972).
6. A. R. Byrne, "Neutron Activation Analysis of Tin in Biological Samples by a Rapid Extraction Separation of  $^{123}\text{Sn}$ ," *Radiochem. Radioanal. Letters* 7, 287-293 (1971).
7. I. Sinko and L. Kosta, "The Determination of Pb, Cd, Cu and Zn in Biological Materials by Anodic Stripping Polarography," *Environmental Analytical Chemistry* (1972), in press.



Project Title: DEVELOPMENT AND APPLICATION OF ULTRASENSITIVE MAGNETIC RESONANCE  
METHODS

Principal Investigator: Professor R. Blinc

Institution: Jozef Stefan Institute, University of Ljubljana, Ljubljana

NBS Monitor: Dr. D. H. Reneker

NBS Institute or Center and Division: Institute for Materials Research  
Polymers Division

#### Summary Description of Project Goals

Nuclear-nuclear double resonance methods were developed more than a decade ago in order to detect NMR and NQR spectra which are too weak to be detected by conventional nuclear magnetic resonance methods. Double resonance techniques have been used to date only in a few cases by physicists and have not yet become a standard tool in the hands of chemists and biologists.

This lack of use is primarily due to the fact that the conditions and detailed procedures under which the various spectra can be collected have not yet been sufficiently delineated. Hence, the technique itself has not yet been fully exploited.

This research has three principal purposes:

1. It is hoped to develop further nuclear double resonance methods in the laboratory frame in order to study low frequency NQR spectra of organic materials in polycrystalline form. They cannot be measured by classical techniques because resonance frequencies are too low or the natural abundance of the nuclei in question is too small. One of the objects of this research is to extend the field of classical  $^{14}\text{N}$ -NQR into the previously inaccessible region of 0.1-- 2 MHz so that the  $^{14}\text{N}$  spectra can be used as an analytical tool for the characterization of various materials.
2. It is intended to use double resonance in the rotating frame for the study of ferroelectric phase transitions. The great sensitivity of pulsed, nuclear-double-resonance techniques in the rotating frame enables

\* The alphanumeric designation refers to the NBS Program Structure (see Appendix II).

one to study the local properties of phase transitions by measuring the NMR spectra of all magnetically active nuclei in the system. The information obtained in this way should significantly enhance our knowledge of the structural changes and the lattice dynamics of ferroelectric phase transitions.

3. It is planned to develop NMR spin-echo methods to measure the translational self-diffusion coefficients in systems where normal NMR spin-echo methods cannot be applied because spin-spin relaxation times are too short.

#### Results and Implications to Date

In the first year of work on this project the research was directed along two lines:

1.  $^{14}\text{N}$  NQR spectra have been determined by level-crossing double resonance in the laboratory frame of reference. This method can be applied as a very high-sensitivity method when there are two spin species in the sample, one of which has a strong resonance signal (which is easily observable by ordinary techniques) whereas the other (which has a quadrupole moment in addition to a magnetic moment) may not be abundant enough or may have a resonance frequency that is too low to be detectable in a conventional NQR or NMR experiment. The method extends the field of classical NQR into the previously inaccessible region of 0.1 - 2 MHz. A spectrometer was constructed, and the  $^{14}\text{N}$  spectra of guanine, thymine, uracil, and cytosine as well as of glycine, proline, cistine, cysteine, methionine, and hystidine have been measured.
2. Double resonance has been used in the rotating frame for the study of ferroelectric phase transitions. The technique of nuclear-magnetic, pulsed double resonance in the rotating frame has been used to study the changes in the  $^{14}\text{N}$  quadrupole interactions in a tri-glycine sulfate (TGS) single crystal on going from the paraelectric to the ferroelectric phase. The  $^{14}\text{N}$  electric-field-gradient (EFG) tensors have been determined above as well as below  $T_c$ . The results show that the phase transition in TGS is of the order-disorder type as far as the glycine groups are concerned. The same technique has been used to determine the EFG tensors at the  $^{14}\text{N}$  sites in paraelectric  $(\text{NH}_4)_2\text{SO}_4$  and the  $^9\text{Be}$  sites in  $(\text{NH}_4)_2\text{BeF}_4$ . The results show that the  $\text{NH}_4$  as well as the  $\text{BeF}_4$

ions are highly distorted and that both of them take part in the ferroelectric phase transition.

In the future these studies are to continue along the above lines. In addition, a start is to be made to develop NMR methods for the determination of self-diffusion coefficients in condensed matter where, because of short spin-spin relaxation times, conventional NMR spin echo methods cannot be applied.

The primary objectives will be:

1. In the field of nuclear double resonance in the laboratory frame, the intention is to study low-frequency NQR spectra of polycrystalline organic materials which cannot be measured by classical NQR methods because of the very low resonance frequencies or low natural abundance. Specifically, we intend to study  $^{14}\text{N}$  spectra of all amino acids and nucleic acid bases and to attempt also peptides and proteins. The double resonance spectrometer developed last year is to be made automatic and calibrated, and its range of operation is to be extended down to liquid helium temperatures.
2. In the field of nuclear double resonance in the rotating frame, the structural phase transitions in several hydrogen bonded ferroelectrics is to be studied in such a way that the NMR and NQR spectra of all nuclei in the sample can be determined.
3. With the help of pulsed field gradients inserted so as not to disturb the Waugh cycles, translational self-diffusion coefficients in condensed matter are to be measured. In particular, liquid crystals and adsorbed molecules on surfaces will be investigated where normal NMR spin-echo methods cannot be applied because of short spin-spin relaxation times. Preliminary experiments showed that this is, in fact, possible.

It should be mentioned that this research is part of a long range research program of the Jožef Stefan Institute. About half of its effort has been funded by Institute sources.

#### List of Publications that Resulted from the Project

1. R. Blinc, M. Mali, R. Osredkar, A. Prelesnik, J. Seliger, and I. Zupancic,  $^{14}\text{N}$

- Quadrupole Coupling in Paraelectric  $(\text{NH}_4)_2\text{SO}_4$ ," Chem. Phys. Letters 14, 49 (1972).
2. R. Blinc', M. Mali, R. Osredkar, A. Prelesnik, I. Zupancic', and L. Ehrenberg, "Pulsed Nitrogen-Proton Double Resonance Study of the Ferroelectric Transition in Triglycine Sulfate," Journal of Chem. Phys. 55, 4843 (1971).
  3. R. Blinc', M. Mali, R. Osredkar, A. Prelesnik, J. Seliger, I. Zupancic', and L. Ehrenberg, " $^{14}\text{N}$  NQR Spectroscopy of Some Aminoacids and Nucleic Bases via Double Resonance in the Laboratory Frame," J. Chem. Phys., (to be published).



Project Title: ELECTROCHEMICAL STUDY OF METAL ION-LIGAND INTERACTION IN NONAQUEOUS SOLUTIONS BY INVESTIGATION OF ELECTROCHEMICAL PROPERTY OF LIGAND

Principal Investigator: Professor Ivan Piljac'

Institution: Institute of Inorganic Chemistry, Faculty of Technology  
University of Zagreb

NBS Monitor: Dr. J. K. Taylor

NBS Institute or Center and Division: Institute for Materials Research  
Analytical Chemistry Division

#### Summary Description of Project Goals

These proposed investigations are for the purpose of finding correlation between the strength of metal-ion-ligand interaction and the changes in electrochemical properties of ligands.

Investigations of hydroxy-quinone and of their conjugate base in non-aqueous media will be performed by polarography and other electrochemical methods. Electrochemical measurements will be combined with spectroscopic ones using the thin-layer, optically transparent electrochemical cell.

This is a new approach to the problem of studying metal ion and organic ligand interactions with the effect of coordination reactions of the molecular orbitals in the ligand which can be related to the electrochemical properties (e.g. half-wave potential). In addition, important information can be obtained concerning the ability of the ligand to donate electrons to metal ions in the process of forming chemical bonds, and further, to identify the type of molecular species resulting from such reactions. The kinds of information and chemical data which are expected to be generated in this project will be of fundamental interest in several areas of electrochemical analysis and of importance to scientists working in coordination chemistry, especially those engaged in chemical problems in such fields as coordination polymers and biomedical research involving metal ion-ligand interactions.

#### Results and Implications to Date

This project was officially started in April 1972 and the first progress report will be submitted to the NBS monitor, Dr. John K. Taylor, in January 1973. Preliminary

\* The alphanumeric designation refers to the NBS Program Structure (see Appendix II).

results have already been obtained on the Hydroxy-9,10 Anthraquinone in non-aqueous media (DMF) indicating that the two-electron reduction step of the hydroxy-quinone base is split under the influence of the metal ion (e.g. lithium ), with a shift of the first wave to more positive potential. The task now is to correlate this shift, of about 100mV, with the strength of the chemical bond. In addition, the mechanism of electro-chemical reduction of the organic ligand will have to be elucidated, in order to understand more clearly the nature of the reaction and bonding.

References:

1. I. Piljać and R. W. Murray, J. Chem. SDC, 118, No. 11, 1758-64 (1971).

This publication summarizes work which was the precursor of this project and was supported by funds other than NBS Special Foreign Currency Funds.

Project Title: MASS SPECTROMETRIC STUDIES OF HIGH TEMPERATURE EQUILIBRIA

Principal Investigator: Dr. Kiro F. Zmbov

Institution: Borid Kidrič Institute, Vinca

NBS Monitor: Dr. John W. Hastie

NBS Institute or Center and Division: Institute for Materials Research  
Inorganic Materials Division

#### Summary Description of Project Goals

A mass spectrometer coupled with a Knudsen cell will be used to study high temperature equilibria involving oxyfluorides, oxides and halides of transition metals and other elements. The results will be used to determine bond dissociation energies and ionization potentials of the molecules studied.

#### Results and Implications to Date

The research program in high temperature mass spectrometry has been under way at the Laboratory for Physical Chemistry of Boriš Kidric Institute for several years. Work in the last three years has been concentrated on building the basic equipment; namely, the mass spectrometer and Knudsen cell assembly. All of this work, as well as the research which has been done prior to the availability of NBS contract funds, has been financed by the Boriš Kidric Institute.

The application of mass spectrometry to high temperature chemistry is widely recognized as an extremely valuable technique, especially for systems that are at equilibrium. A large variety of systems have been studied in which many molecules have been identified and their dissociation energies determined using both second and third-law treatments of the experimental data. Alternatively, one may use electron impact appearance potential measurements as an independent source of thermochemical data. This technique has value where the third-law treatment cannot be used owing to large uncertainties in the molecular partition functions.

Most of the mass spectrometric studies made so far have used conventional Nier-type ion sources, in which molecules effusing from the Knudsen cell are ionized by electrons

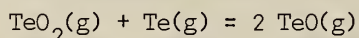
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\* The alphanumeric designation refers to the NBS Program Structure (see Appendix II).

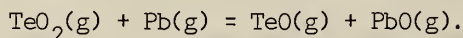
emitted from an incandescent tungsten filament. The energy spread of the electrons is large, and the electron impact data are obtained with an uncertainty of about 0.5 eV (10 kcal). The use of a "monoenergetic" electron beam for ionization of high temperature molecules would provide a more accurate determination of the ionization potentials and bond dissociation energies of these molecules.

During 1972, the following work has been performed on the project:

- a) The design of the Knudsen cell attachment for the mass spectrometer has been completed. Cells of tantalum or graphite have been tested. Calibration of the system with Ag and Tl indicated a pressure sensitivity of the order of  $10^{-7}$  torr, using a cell orifice diameter of 0.1 mm.
- b) An apparatus including the Knudsen cell system and a mass spectrometer (a magnetic sector instrument with a  $90^\circ$  sector, a 20-cm radius of curvature, and an electron-multiplier detector) has been used to study equilibria between  $\text{TeO}_2$  and Te and also Pb in the gas phase. Enthalpies of the following reactions have been measured:

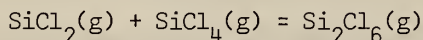


and



From the measured enthalpies and the known heats of formation of  $\text{TeO}_2(\text{g})$  and  $\text{PbO}(\text{g})$ , the heat of atomization of the  $\text{TeO}(\text{g})$  molecule has been determined from the two independent reactions. The results of this investigation are being prepared for publication.

- c) The composition of the gas phase above the reaction mixture  $\text{CuCl}+\text{Si}$  in a copper Knudsen cell has been investigated. From the mass spectra and appearance potentials of the ions, the following "parent" molecules have been identified at temperatures near  $200^\circ\text{C}$ :  $\text{SiCl}_2$ ,  $\text{SiCl}_4$ ,  $\text{Si}_2\text{Cl}_6$ ,  $\text{Si}_3\text{Cl}_8$  and  $\text{Si}_2\text{OCl}_6$ . The ionization potentials of these molecules have been measured, and the reaction



has been studied over a range of temperatures ( $160^\circ - 200^\circ\text{C}$ ).



Equilibrium constants for this reaction have been measured, and the reaction enthalpy has been determined by applying methods based on the second law of thermodynamics. These data were used to calculate a value for the Si-Si bond dissociation energy in the silicon hexachloride molecule of  $71 \pm 4$  Kcal/mol. Considerable controversy exists concerning this bond energy; different authors have reported values ranging from 50 to 80 Kcal/mol.

The results of this investigation were reported at the 15th Yugoslav Congress of Pure and Applied Chemistry, Ljubljana, June 1972. The results should be considered as preliminary, and calculations based on the third law of thermodynamics should be performed for comparison with the results obtained by the second-law procedure. For this purpose free energy functions are needed, and these are being determined in collaboration with the NBS monitor, Dr. J. W. Hastie.

- d) A trochoidal electron monochromator (TEM) has been designed and installed in the mass spectrometer. The initial design has been modified in order to shield the electron beam from stray magnetic fields. The electron stopping curves have been measured by the retarding potential method, and the energy spread of the electron beam has been measured at both the entrance and exit of the monochromator. Beams with an energy spread of 0.04 eV were obtained, but their intensity of about  $10^{-8}$  A was rather small. This leads to too few ions being produced for analysis.

These results indicate a need for further technical improvements of the TEM such as a better alignment of the electrode system. Improvement of the spectrometer detection sensitivity is anticipated by replacing the standard Cu-Be multiplier by a "Channeltron", thus increasing the sensitivity by two to three orders of magnitude. With these improvements, we hope to obtain a system of the highest possible quality for precise measurements of the ionization potentials and bond dissociation energies of high temperature-molecules.



Project Title: STARK BROADENING OF SPECTRAL LINES IN PLASMAS

Principal Investigator: Dr. N. Konjevic

Institution: Institute of Physics, University of Belgrade, Belgrade

NBS Monitor: Dr. W. L. Wiese

NBS Institute or Center and Division: Institute for Basic Standards  
Optical Physics Division

Summary Description of Project Goals

This project concerns itself with the study of the Stark broadening and shift of some prominent spectral lines of lighter elements in plasmas. There is a considerable interest in investigations of Stark broadening of isolated spectral lines in plasmas because of several discrepancies between experiment and theory. In addition, from the plasma diagnostic point of view, there is a constant interest in the line broadening data for lines of different elements including ionic species since these may be applied for determinations of the plasma electron density. Therefore, a number of experimental and theoretical papers have been recently devoted to the investigation of Stark broadening and the shift of isolated neutral and ion lines. However, most of these investigations are related to the study of the broadening parameters of lines belonging to hydrogen and helium. This is mainly due to the following practical plasma diagnostic conditions: a) stellar atmospheres consist mainly of hydrogen atoms or ions with an admixture of helium and traces of other elements and b) in a number of laboratory experiments these gases are present; also, atomic wave functions are best known for these elements, thereby aiding in the testing of the basic theoretical approach.

However, only a few theoretical papers treat Stark broadening of more complex atoms and ions, and these results represent, in general, further refinements of the Griem-Kolb-Shen-Oertel theory. Relatively few experimental papers investigate broadening of the lines of these elements; therefore, most atoms have not been investigated at all.

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\* The alphanumeric designation refers to the NBS Program Structure (see Appendix II).



The aim of this project is to provide data on the Stark broadening and shift of neutral and ion lines for some more complex atomic systems. Fluorine, aluminum and magnesium are selected, mainly for technical reasons. In order to find optimal experimental conditions for excitation of spectral lines, this work involves a thorough investigation of various plasma sources and plasma diagnostic techniques.

### Results and Implications to Date

At the time of writing this report, determination of Stark widths and shifts of some prominent spectral lines of fluorine I has been completed in a zeta-pinch plasma in dischlorodifluoromethane. The electron density was determined by laser interferometry, while the electron temperature was measured from relative intensities of Cl II lines. Temperatures were in the range 19,000 - 36,200 K; electron densities varied from 0.71 to  $1.43 \times 10^{17} \text{ cm}^{-3}$ . Experimentally determined half-halfwidths in Å units normalized to the electron density  $10^{17} \text{ cm}^{-3}$  of ten F I lines at various temperatures are given in Table 1, along with estimated uncertainties (caused by the error in determination of electron density and Stark profiles). The tabulated data are compared with values calculated by Griem.<sup>(1)</sup> The ratios of experimental line shifts to half-halfwidths of five fluorine multiplets at various temperatures are listed in Table 2 and compared with theoretically predicted values.<sup>(1)</sup> From the comparison of experimental and theoretical values in Tables 1 and 2, it is shown a) that experimental widths are systematically smaller and the degree of the discrepancy increases with temperature, b) that line shifts are also smaller than predicted with the exception of multiplets 5 and 6 and the shifts are of the opposite sign than theoretically predicted, and c) that experimental shift-to-width ratios decrease with the temperature but less than theoretically expected.

Finally, attention is drawn, in general, to the systematic discrepancy between the experimental results for neutral atom lines and the theory. The magnitude of this disagreement is related to the relative position of energy levels of the spectral line to the ionization limit.

#### References:

1. H. R. Griem, "Plasma Spectroscopy," New York: McGraw-Hill (1964).

List of Publications that Resulted from the Project

1. N. Konjević, M. Platiša and M. Popović, "Stark Broadening and Shift of Fluorine I Lines," in press (to be published in Z. f. Phys.).

TABLE 1

Experimental half-widths  $W(\text{\AA})$  at  $10^{17}$  electrons  $\text{cm}^{-3}$  of fluorine I lines at various electron temperatures compared with theoretical values computed by Griem<sup>1</sup> for the same experimental conditions.

Temperatures	19100 K	23650 K	28200 K	29400 K	36200 K
Mult. No 1					
7331.9	0.15+0.03	0.15+0.03	0.16+0.03	0.16+0.02	0.16+0.02
7398.7	0.16+0.03		0.16+0.03		
Theory	0.28	0.31	0.33	0.34	0.36
-----					
Mult. No 2					
6902.5	0.17+0.03	0.17+0.03	0.17+0.02	0.18+0.02	0.18+0.02
6856.0	0.17+0.03	0.18+0.03	0.18+0.02	0.18+0.02	0.18+0.02
Theory	0.28	0.31	0.33	0.34	0.36
-----					
Mult. No 3					
6239.6	0.16+0.03	0.16+0.03	0.17+0.03	0.17+0.02	
6413.6		0.16+0.03	0.16+0.03	0.16+0.03	0.17+0.02
6348.5	0.16+0.03	0.17+0.03			0.17+0.02
Theory	0.28	0.31	0.33	0.34	0.36
-----					
Mult. No 5					
7311.0		0.12+0.03	0.12+0.03		0.13+0.03
Theory		0.15	0.15		0.16
-----					
Mult. No 6					
7037.5		0.11+0.03	0.12+0.03		
7127.9			0.12+0.03		
Theory		0.13	0.14		

TABLE 2

Measured shift-to-half-halfwidth ratios of fluorine I lines compared with theoretical values.<sup>1</sup>

Temperatures	19100 K	23650 K	28200 K	29400 K	36200 K
Mult. No 1					
7331.9		0.73+0.22	0.69+0.19	0.61+0.15	
7398.7	0.68+0.20		0.65+0.18		
Theory	0.98 <sup>-</sup>	0.89	0.82 <sup>-</sup>	0.80	
-----					
Mult. No 2					
6902.5	0.72+0.21	0.61+0.18	0.72+0.21	0.62+0.17	0.59+0.15
6856.0	0.86+0.26	0.72+0.22	0.67+0.20	0.74+0.20	0.77+0.19
Theory	1.05 <sup>-</sup>	0.95 <sup>-</sup>	0.87 <sup>-</sup>	0.85 <sup>-</sup>	0.76 <sup>-</sup>
-----					
Mult. No 3					
6239.6	0.89+0.26	0.81+0.24	0.82+0.22	0.74+0.18	
6413.6		0.85+0.24	0.81+0.20	0.78+0.18	0.77+0.17
Theory	1.05	0.95 <sup>-</sup>	0.87 <sup>-</sup>	0.85 <sup>-</sup>	0.76 <sup>-</sup>
-----					
Mult. No 5					
7311.0		1.00+0.35	1.08+0.32		0.88+0.28
Theory*		-0.98 <sup>-</sup>	-0.93 <sup>-</sup>		-0.85 <sup>-</sup>
-----					
Mult. No 6					
7037.5		1.27+0.37	1.08+0.35		
7127.9			1.04+0.33		
Theory*		-0.98	-0.93 <sup>-</sup>		
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\* Note: A positive shift is red.

Project Title: PART II, ELECTRON-ATOM ELASTIC SCATTERING

Principal Investigator: Dr. M. Kurepa

Institution: Institute of Physics, Belgrade University

NBS Monitor: Dr. C. Kuyatt

NBS Institute or Center and Division: Institute for Basic Standards  
Optical Physics Division

Summary Description of Project Goals

The goal of this project is to measure the absolute differential cross-section of elastically scattered electrons from inert gas atoms. At present, the energy range of study is from 60 to 200 eV. This is to be extended to cover the energy range from 10 to 800 eV. The purpose of this investigation is:

- (1) to obtain a known standard for inelastic differential cross section determination;
- (2) to check the validity of the Born approximation (theory) at higher energies; and
- (3) to calculate the phase shifts for scattering from which the atomic scattering potential can be obtained.

These data will be applicable for the determination of diffusion cross-section of electrons contained in a plasma.

This project is in effect a continuation of research which began two years ago. Apparatus now exists for measurements of elastically scattered electrons. The list of publications that follow summarizes the results obtained before the NBS/SFCP funded project began.

In order to continue the investigations and meet the project goals, it will be necessary to improve the electronics associated with the stability of the beam intensity and energy, as well as the detection of very low scattered electron intensities.

References:

1. L. Vuskovic, "Angular Scattering of Low Energy Electrons on Atoms," PhD Thesis,

\* The alphanumeric designation refers to the NBS Program Structure (see Appendix II).



PMF, 1972, Beograd University.

2. L. Vušković, "Differential Cross Sections for Elastic Scattering of Electrons on Atoms," Invited Lecture on the VI Yugoslav Summer School on the Ionized Gases, Split, 1972. (To be published in the Proceedings of the Summer School.)
3. M. Kurepa, L. Vušković and S. Kalezić, "Absolute Differential Cross Sections of Electrons Elastically Scattered on Neon Atom," Proc. of the VI Yugoslav Symposium on the Physics of Ionized Gases, 1972, page 17.
4. L. Vušković and M. Kurepa, "Absolute Differential Cross Sections of Electrons Energies 100, 150 and 200 eV Elastically Scattered on Helium Atom" (in Russian), Svesojuznaja Konferencija po Atomnihj Elektronih Stolknovenjij, Uzgorod, 1972, SSSR.
5. M. Kurepa and L. Vusković, "Total Cross Section for Elastic Scattering of Electrons at Energies from 60-150 eV on Argon Atoms," (in Russian), Svesojuznaja Konferencija Do Atomnihj Elektronih Stolknovenjij, Uzgorod, 1972,SSSR .

#### Results and Implications to Date

This grant was activated recently. No direct results can therefore be claimed at this time.

Project Title: MEASUREMENT OF f-VALUES IN THE ULTRAVIOLET AND VISIBLE REGION AND STUDY  
OF CONTINUOUS EMISSION OF HYDROGEN NEAR THE BALMER LIMIT

Principal Investigators: Dr. D. Müller and Dr. V. Vujnović

Institution: Institute of Physics, University of Zagreb

NBS Monitor: Dr. W. L. Wiese

NBS Institute or Center and Division: Institute for Basic Standards  
Optical Physics Division

Summary Description of Project Goals

This project, which is intended to be active over three years, can be divided into two parts, one experimental and one theoretical. The primary aim of the experimental investigation is to provide the National Standard Reference Data System (NSRDS) with data on atomic transition probabilities. These data are not presently as accurate as is required for diagnostics in plasma spectroscopy used in determinations of plasma composition and state. Measurements will be done, first of all, for absolute oscillator strengths of atomic and ionic lines of phosphorous in the visible region. Such experiments have not been reported on previously. In addition, by employing an improved experimental technique, oscillator strengths of lines in the vacuum ultraviolet (VUV) are to be determined for some lines which have been observed for certain elements but whose oscillator strengths have not been measured quantitatively.

In the theoretical part of the project, a study will be undertaken in an attempt to explain the spectral distribution of continuous emission from hydrogen near the Balmer limit. The approach to this problem will be to compare a theoretical recombination continuum with experimentally acquired data from the NBS Plasma Spectroscopy Section or with the other data of equal accuracy. The purpose of this exercise is to investigate and explain quantitatively the discrepancies between experiment and theory. Such results are important in the explanation of plasma physics phenomena and in the interpretation of astronomical observations since hydrogen is the most abundant atom involved.

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\* The alphanumeric designation refers to the NBS Program Structure (see Appendix II).

As of the time of preparation of this report, funds to support the project have not been received. However, on their own initiative, the principal investigators have begun this project. The equipment has been assembled except for the one meter, visible-region monochromometer which is to be purchased from an American manufacturer at a cost of about \$18,000. The order for this instrument is ready to be placed immediately.

Some of the problems associated with the project have been investigated and are discussed in the publications listed below:

References:

1. D. Müller, "Vacuum UV Emission of High Density Plasma, generated in a Wall-stabilized Arc," Proc. International Summer School of Ionized Gases, 694 (1970).
2. K. Acinger, D. Müller, G. Pichler, A. Mejaški-Isnejc, and V. Vujnović, "Measurements on a wall-stabilized cascade Argon Arc," Fizika 2 Suppl, 1, 85 (1970).
3. K. Acinger, G. Pichler, V. Vujnović and D. Vukicević, "Measurement of Line-oscillator Strengths of Spectral Lines Which are not Optically Thin," Proc. 6th Yug. Symp. on Phys. of Ionized Gases, Split, 209 (1972).
4. D. Müller, G. Pichler and C. Vadla, "An Estimation of the Stark Half-width of the CI 2478 Spectral Line," Proc. 6th Yugoslavia Symp. on Phys. of Ionized Gases, Split, 255 (1972).
5. V. Vujnović, "The Statistical Recombination Continuum and its Relation to the Lowering of Ionization Energy," J. Qualitative Rad. Transfer 10, 929 (1970).



Project Title: PART I, ELECTRON-ATOM EXCITATION CROSS-SECTION MEASUREMENTS

Principal Investigator: Dr. Vladeta Urosevic'

Institution: Institute of Physics, Belgrade University

NBS Monitor: Dr. G. Dunn

NBS Institute or Center and Division: Joint Institute for Laboratory Astrophysics

### Summary Description of Project Goals

This project is to measure a few key line excitation cross-sections with the highest attainable accuracy. The goal is to establish an absolute line excitation cross-section standard. There now exist large disagreements in line excitation cross-section data. A suggested approach to clarify these disagreements is to establish an absolute cross-section standard. With this standard, an excitation apparatus could be calibrated for relative measurements of any unknown excitation line cross section. After such a calibration, using only a pressure gauge independent of the nature of the gas, such an apparatus would be capable of accurate measurements of an unknown excitation line cross-section. This means that it will not be necessary that every laboratory have absolute pressure, electron current, and radiation standards required for absolute excitation cross section work.

In order to verify and establish the use of such an absolute standard, the principal investigator and his colleagues have designed two different pieces of experimental equipment. The first device (apparature A - POBA) is to demonstrate the proper method of employing the absolute standard to calibrate relative measurements, and a second experimental device (apparatus B) is to be used for the determination of the absolute standard.

To check this suggested approach, one unknown line in Ne (at  $\lambda(\text{\AA}) = 4827.5 + 4827.3$ ) has been measured and compared with the best reported He line cross-section measurement (according to Kiefer's Atomic Data (1969)). The results of this investigation are given in reference 1.

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\* The alphanumeric designation refers to the NBS Program Structure (see Appendix II).

In 1972 apparatus A (POBA) was completed. A necessary by-product of this work was the design and development of special electron optics required for electron excitation measurements. The four-element cylindrical lens designed for this use is discussed in reference 2. This work has been published (ref. 2).

Another accomplishment was the radiometric calibration of the detection system for both devices employing an independent approach. In the calibration two tungsten Osram lamps provided secondary standards.

The Institute of Physics has completely financed apparatus A. In addition, the Institute has appropriated about \$12,000 for the purchase of an ultrahigh vacuum chamber, the necessary ultrahigh vacuum (to  $10^{-11}$  torr) pumping system, and an earth's magnetic-field compensator. These are all essential for apparatus B.

Recent publications of the group may be consulted from details (refs. 4, 5 and 6).

#### Results and Implications to Date

This grant was activated recently. No direct results can therefore be claimed at this time.

#### References:

1. M. D. Tasic, J. M. Kurepa and V. M. Urosević, "The use of the known Helium Line Cross Section for the Investigation of an Unknown Transition in Neon." Proc. of VI Yugoslav Symposium of Ionized Gases, Split (1972).
2. M. V. Kurepa, M. D. Tasić and J. M. Kurepa, "Investigation of the Characteristics of Four Element Cylindrical Electrostatic Lenses." Proc. of XVI Yugoslav ETAN Conference, Velenje (1972).
3. M. D. Moracić-Tasić, "The Absolute Measurement of Spectral Radiance of Low Intensity Light Sources by the Single Photon Counting Method." Thesis for Master of Science Degree, PMF, Beograd (1972).
4. J. M. Kurepa, "The Structure Investigations of the Electron Excitation Functions in Helium near the Threshold," Thesis, PMF, Beograd (1972).
5. D.W.O. Heddle, R.G.W. Keesing and J. M. Kurepa, "High Resolution Studies of Electron Excitation I. The 4S States of Helium and the Energy Scale," submitted to Journal of Phys. B. (Great Britain).

6. D.W.O. Heddle and J. M. Kurepa, "The Near Threshold Behaviour of Electron Excitation Functions in Helium." Proc. of Invited paper for VI Yugoslav Symposium on Physics of Ionized Gases, Split (1972), to be published.





Project Title: BULK COMPOSITION AND SURFACE PROPERTIES OF GLASS

Principal Investigator: Dr. Velimir Pravdić

Institution: Ruđer Bošković Institute, Zagreb

NBS Monitor: Dr. Wolfgang Haller

NBS Institute or Center and Division: Institute for Materials Research  
Inorganic Materials Division

#### Summary Description of Project Goals

The objectives of this Project have been to study the surfaces of glasses of well defined bulk composition and to find correlations between selected surface parameters and the bulk composition.

There is a lack of full understanding of the extent of such correlations. Furthermore, chemical or physical treatments of the surface of a glass of given bulk composition could impart a variety of desired characteristics. A fuller understanding of these correlations could be of technological importance wherever glass is used.

The properties to be studied include the surface charge distribution as measured by electrokinetic techniques, by ESR hyperfine splitting, and by heats-of-immersion calorimetric measurements. Thus charge densities in the surface layers could be determined.

#### Results and Implications to Date

The collaboration with the NBS monitor is a close one. Mutual visits have occurred at both institutions. Well characterized samples have been supplied by the NBS Glass Section. These are studied at the Electrochemistry Laboratory of the Ruđer Bošković Institute using techniques which have been a speciality of this research group for some time. The NBS materials are thus further characterized for use in other experiments or for possible technological exploitation. As results accumulate, further experimental strategy is planned jointly between NBS and Ruđer Bošković Institute.

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\* The alphanumeric designation refers to the NBS Program Structure (see Appendix II).

The specific experimental activity at the Rüdger Bošković Institute included the study of ionic movement along the surfaces of glasses, either in powdered or in capillary form, using electrokinetic techniques, namely streaming potential, streaming current, and surface conductance measurements. Application of the diffuse double layer theory permits the calculation of charge densities in the liquid at the "slipping" plane (the mobile/immobile interface).

After the project had started a highly relevant new technique was invented<sup>1</sup>. Measurements of the ESR hyperfine splitting spectrum for a suitable labeled atom ( $^{107}\text{Ag}$ ), adsorbed at the surface, can be used to estimate the contribution of the electrostatic field to the total interaction energy. From this, the local charge density which the labeled atom "sees" at the surface can be derived. Different treatment of surfaces in the adsorption progress with ionic silver and a subsequent irradiation procedure to produce atomic silver, gives adequate knowledge on the actual location of the labeled atoms in the surface layers. The location of the silver atom has been found in the swollen gel layer, between the solid surface proper and the liquid or vapor interface.

Even more important, the energetics of the actual solid surface of some glasses have been studied by heat-of-immersion calorimetry. A series of liquids with different dipole moments has been used, and the contribution of polar forces to the total interfacial energy has been calculated.

Glasses studied so far have been the commercially available brands of pure fused silica, doped silica, Vycor, Pyrex (7740), and a selection of controlled-pore glasses produced in the NBS laboratories.

The results obtained are exemplified by a case study of one of the controlled-pore glasses (NBS product Code WHB 380 BS, 225 nm average pore size, and  $10.4\text{m}^2/\text{g}$  nitrogen surface area). The following distribution of charge densities has been found:

- |  |  |
|--|--|
| i) From the heats-of-immersion of vacuum-heated (300° C) samples:<br>Fraction of energy of polar forces to total interfacial energy: 0.35  | 150 x 10 <sup>12</sup>                           |
| ii) From ESR hyperfine splitting for <sup>107</sup> Ag <sup>0</sup><br>calculated contribution of electrostatic interaction:<br>- simple adsorbed Ag <sup>0</sup><br>- "caged" Ag <sup>0</sup> upon vacuum heating | 6.6 x 10 <sup>12</sup><br>5.5 x 10 <sup>12</sup> |
| iii) From electrokinetic streaming current in a 1:1 electrolyte<br>(HCl + NaCl, 10 <sup>-4</sup> moles/l)  | 2.7 x 10 <sup>12</sup>                           |

Data obtained for other glasses lead to similar results. It is therefore concluded that minor variations in bulk composition (doping) will not strongly influence the charge distribution in the outer layer (electrokinetics). Differences, if any, would be within the experimental error. Calorimetric techniques bear more promise in relating the surface charge densities with bulk properties. These techniques provide complementary data to studies involving charge, or ion, transport across the interface. Potentials developed in glass membranes of ion-specific electrodes provide such an example. In addition, the energetics of adsorption of various species at glass surfaces would be a measure of the technological suitability of certain glasses for special requirements.

#### References:

1. G. L. Gardner, E. J. Casey and C.W.M. Grant, J. Phys. Chem., 74, 3273 (1970).

#### List of Publications that Resulted from the Project

1. J. Jednačak, M. Hudomalj, V. Pravdić and W. Haller, "Electrokinetics of Glass Surfaces, I. Charge Densities at the Solid/Gas and the Solid/Liquid Interfaces of Porous Glasses," Croat. Chem. Acta 44, 149 (1972).
2. J. Jednačak and V. Pravdić, "Electrokinetics of Glass Surfaces," II., Paper presented at the 3rd Yugoslav Congress for Pure and Applied Chemistry, Ljubljana (June 1972). Full paper prepared for publication.
3. M. Hudomalj, K. Adamić and V. Pravdić, "Investigation into the Electric Field at Glass Surfaces by ESR," *ibidem*. Full paper in preparation.

Project Title: SYNTHESIS AND PROPERTIES OF FLUORIDE COMPLEXES

Principal Investigator: Professor Dr. J. Slivnik

Institution: Jožef Stefan Institute, Ljubljana

NBS Monitor: Dr. T. D. Coyle

NBS Institute or Center and Division: Institute for Materials Research  
Inorganic Materials Division

#### Summary Description of Project Goals

This work deals with investigations of reactions of transition-element fluorides with anhydrous hydrazine; the synthesis and isolation of hydrazinium fluorometallates containing first row transition elements; and the preparation and characterization of xenon difluoride and fluorides of the first row transition series; for the following purposes:

1. To develop new or improved methods of chemical synthesis for important inorganic fluorine containing compounds;
2. To advance the techniques of sample purification, particularly where improvements in sample purity will enhance the growth of more perfect single crystals, and
3. To accurately determine the chemical and physical properties of selected fluorine containing compounds where such data will contribute to the understanding and behavior (reactions) of such compounds.

#### Results and Implications to Date

Although this project was originally started with Special Foreign Currency Program funds in July 1972, work on the fluoride compounds related to this investigation was begun at least a year earlier, entirely on funds from the Jožef Stefan Institute. Thus, there has been, and continues to be significant cost-sharing on this project. In fact, the first progress report submitted to NBS on the synthesis of and properties of fluoride complexes was dated February 15, 1972, more than four months before the official transfer of funds to the project leader, Dr. J. Slivnik.

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\* The alphanumeric designation refers to the NBS Program Structure (see Appendix II).



This report indicated progress in the following areas:

- (a) Investigations of reactions of fluorine-containing compounds of the transition elements with anhydrous hydrazine.

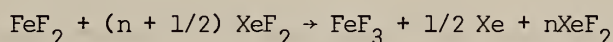
Zirconium tetrafluoride-dihydrazine ( $\text{ZrF}_4 \cdot 2\text{N}_2\text{H}_4$ ) has been prepared. Infra-red analysis and the study of thermal decomposition have been undertaken. Additional compounds of Hafnium ( $3\text{HfF}_4 \cdot 5\text{N}_2\text{H}_4$ ) and cerium ( $2\text{CeF}_4 \cdot \text{N}_2\text{H}_4$ ) have been synthesized and isolated. Their properties are now being studied. These compounds were obtained at a purity of 99.0-99.5 per cent.

- (b) Synthesis of Hydrazinium Fluorometallates of first row transition elements.

Preliminary work has led to the preparation of fluorine compounds of manganese and iron. Infrared spectroscopic data and x-ray diffraction data will be obtained on these compounds to determine their structure and exact chemical formula.

- (c) Synthesis of compounds formed from xenon difluoride and first row transition element fluorides.

The reaction of iron difluoride and xenon difluoride at  $120^\circ$  is as follows:



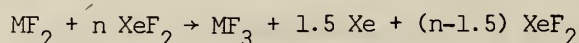
where  $n > 2$

A second report has been completed and contains additional results on topics (a), (b) and (c), above. First it was found that  $\text{CoF}_3$  and some other high-valent fluorides reacted explosively with liquid anhydrous hydrazine and that in other cases reduction of the higher valent fluorine compounds occurred. Thus the work described in the second report was confined to compounds which did not undergo oxidation or reduction reactions with hydrazine, namely;  $\text{ZrF}_4$ ,  $\text{HfF}_4$  and  $\text{CeF}_4$  from group 4b;  $\text{ZnF}_2$  and  $\text{CdF}_2$  from group 2b; and  $\text{MnF}_2$  plus  $\text{NiF}_2$ . The details of the preparative procedures are given as are the chemical analyses of the tetrafluoro and difluoro compounds.

The reaction of metal fluorides with anhydrous hydrazine to give metal fluoride-hydrazine adducts was carried out in an all glass system at room temperature using a large excess of hydrazine, employing a tensimetric technique. The vapor pressure in the reaction vessel was measured with a mercury manometer yielding data on the equilibrium pressure at various metal fluoride to hydrazine mole ratios. The infrared spectra of the compounds were obtained with a Zeiss UR-20 spectrophotometer, using the Nujol Mull technique. Thermal analyses of the samples with a Mettler thermal analyzer (heating rate 6°C/min., sample weight 100 mg, and argon atmosphere with a flow rate of 5 liters per hour) yielded data on the temperatures identified with endothermic and exothermic reactions.

The work on the first row transition metal compounds discussed in the section dealing with the first report has now been extended to compounds containing cobalt and nickel, such compounds having the general formula  $N_2H_6MF_n$  and  $N_2H_5MF_n$  where  $n = 4-6$ . Furthermore, some preliminary results have been obtained on copper compounds.

Finally, reactions between xenon difluoride and first row transition metal fluorides were carried out in a more detailed and systematic manner. Because nickel and copper difluoride did not react with xenon difluoride, the bulk of the investigation was related to reactions involving powders of iron, cobalt and chromium fluorides with xenon difluoride at 200°C to form the trifluoride shown below:



where  $n \geq 4$

No evidence was obtained of a reaction between the trifluoride and the xenon difluoride to give an addition compound..

Several implications of this work are given here. First the thermal decomposition of a number of the fluoro-compounds lead to very pure fluoride compounds which can be used as starting materials for growing pure single crystals. The importance of this subject was treated in a recent paper presented at the 4th European Symposium on Fluorine Chemistry (S.V. Petrov, Institute of Physical Problems, Moscow, "Synthesis of  $MnF_2$ ,  $FeF_2$ ,  $CoF_2$  and  $NiF_2$  for the Growing of Single Crystals"). Secondly, the difluorides of the first row transition metals are reported to have useful applications in microelectronics.

It should also be pointed out that, in addition to generating data and information on the chemistry of fluoro-compounds, this project is also promoting the development of methods of preparation, purification and characterization of fluorine compounds.

List of Publications that Resulted from this Project

1. P. Glavić and J. Slivnik, "Reactions of Transition Metal Fluorides with Anhydrous Hydrazine (I)" (in preparation); presented at the 4th European Symposium on Fluorine Chemistry (1972).
2. B. Zemva and J. Slivnik, "Some Reactions of Chromium, Iron, and Cobalt Fluorides with Xenon Difluoride and Hexafluoride" (in preparation); presented at the 4th European Symposium on Fluorine Chemistry (1972).





Project Title: PATTERN RECOGNITION AND PROCESSING

Principal Investigator: Dr. Vladimir Bonacic'

Institution: Ruđer Bošković Institute, Zagreb

NBS Monitor: T. N. Pyke, Jr.

NBS Institute or Center and Division: Center for Computer Sciences and Technology  
Information Processing Technology Division

#### Summary Description of Project Goals

Research will be conducted in the area of pattern recognition utilizing techniques for pseudo-random digital transformation of data from biological and nuclear experiments. Various techniques for identifying patterns in data shall be studied and compared.

#### Results and Implications to Date

New techniques for optimal coding of experimental data by means of pseudo-random digital transformations have been developed, and the total range of pseudo-random digital transformers have been classified. In addition, some useful properties of these transformers have been identified, especially relative to potential convenient implementation of these transformations for storing and analyzing experimental data.

Pseudo-random digital transformers have been classified into linear and non-linear systems. Further classification of these two categories has been based upon the complexity of connections within the systems. The relationship among particular kinds of linear systems has been determined and is expressed in the form of a theorem about the interrelationships of linear pseudo-random digital transformers. This theorem opens the possibilities of utilizing parallel rather than serial techniques in implementing such transformers. From the point of view of on-line application, where computation is performed in real time, this result is an important contribution in shortening transformation time for data display. Furthermore, it has been shown that parallel equivalents of linear and non-linear systems are only slightly more complex in hardware implementation than serial ones.

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\* The alphanumeric designation refers to the NBS Program Structure (see Appendix II).

A pattern test has been introduced in a search for the properties of pseudo-random sequences obtained by digital transformers. This test is based upon the visual, two-dimensional display of pseudo-random number sequences. Testing using this technique has shown that the output pseudo-random sequences associated with pseudo-random digital transformations show extraordinary regularity for linear transformations, while for non-linear ones randomness is observed. This computational-coupled, visual technique for observing patterns in pseudo-random sequences has become a useful tool in testing for pseudo-random properties in such sequences.

List of Publications that Resulted from the Project

1. M. Cimerman and V. Bonacić, "On the Criteria for Classifying the Pseudo-Random Digital Transformers," (prepared for publication).
2. V. Bonacić and M. Cimerman, "The Pattern-Testing of Pseudo-Random Digital Transformers," (to be published).
3. M. Cimerman and V. Bonacic, "On the Relations Among Linear Pseudo-Random Digital Transformers," (to be published).

Project Title: A MODULAR COMPUTER SYSTEM FOR REAL-TIME DATA REDUCTION AND  
CONTROL IN BIOMEDICAL EXPERIMENTS

Principal Investigator: Professor Zvonimir Damjanović

Institution: Multidisciplinary Centre of Belgrade University

NBS Monitor: T. N. Pyke, Jr.

NBS Institute or Center and Division: Institute for Computer Sciences and Technology  
Information Processing Technology Division

Summary Description of Project Goals

A small modular computer system will be designed for real-time data acquisition and control of biomedical experiments. This configuration will be implemented and used as testbed for studying the experimental computer interface and for developing new techniques that give the experimenter flexible command over the apparatus without the need for him to become familiar with the detailed operation of the supporting computer. During control of a biomedical experiment, it is desired that the human operator have the necessary but minimum interaction with the computer-experiment complex.

The main effort in the first year of the project is concentrated on the interfaces between the experiment, computer, and experimenter. Of particular interest is the definition of an intermediate level experimenter's programming language that will be more suitable for biomedical research and other applications than existing programming languages. In order to achieve these goals, the following activities are being pursued within the project:

- (a) Examination of several representative biomedical experiments in which computer application is indispensable
- (b) Identification of characteristic signal processing methods in biomedical experiments
- (c) Study of input-output devices necessary for the realization of mutual communication in the system composed of man, computer and the biological object

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\* The alphanumeric designation refers to the NBS Program Structure (see Appendix II).

- (d) Specification of requirements that have to be met by the programming language intended for computer control of biomedical experiments
- (e) Definition of the programming language for computer control of biomedical experiments
- (f) Illustrative application of the programming language in several experiments.

#### Results and Implications to Date

The following three typical biomedical experiments have been studied so far:

- (1) The light controlled electrical activity of the ganglion of APLYSIA.
- (2) The ringing response of Algal cells
- (3) An experiment to study the ability of the muscular system of a human operator to produce command signals. This experiment was performed with the aid of a computer programmed in assembly language.

Other activities within the project are in progress.



Project Title: RADIATION DOSIMETRY AND RADIATION CHEMISTRY

Principal Investigator: Dr. I. Draganić

Institution: Boris Kidrič Institute of Nuclear Sciences, Vinca

NBS Monitor: William L. McLaughlin

NBS Institute or Center and Division: Institute for Basic Standards  
Center for Radiation Research, Applied  
Radiation Division

### Summary Description of Project Goals

Radiation dosimetry and radiation chemistry studies will be made using very short electron pulses ( $\sim 10$  ns) of very high intensity ( $\sim 10^{10} \text{ J cm}^{-2} \text{ s}^{-1}$ ). The studies will include: Absolute calorimetric measurement of total beam energy and partial beam absorption in thin foils; relative dosimetry for dose distribution measurements, using chemical and physical methods; chemical studies on suitable dosimetry systems, including radiochromic dyes, thiocyanides, ferrocyanide, and others; pulse radiolysis studies of chemical reactions of transient chemical species. These studies are expected to provide important information on the behavior of dosimetry systems currently being used to measure radiation absorbed doses and dose distributions in intense radiation fields.

### Results and Implications to Date

A total absorption calorimeter was designed for measuring total electron beam energy from a continuous, scanned, or pulsed electron beam. Tests have shown that, even for pulses as short as tens of nanoseconds and of the order of  $10^{10} \text{ J cm}^{-2} \text{ s}^{-1}$  energy flux density, an accurate measurement of total energy fluence was possible with a reproducibility of less than  $\pm 4$  percent. This instrument has been used to calibrate the response of thin radiochromic-dye dosimeters to various intense electron beams. This calibration enabled the dosimeters to be used interchangeably in pulsed, scanned, or continuous electron beams varying in energy from 0.4 to 10 MeV and in a dose rate range from  $10^6$  to  $10^{13}$  rads per second.

The calorimeter was of a differential quasiadiabatic type, consisting of a totally absorbing aluminum absorber adjacent to, but insulated from, a reference calorimetric body.

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\* The alphanumeric designation refers to the NBS Program Structure (see Appendix II).

The system is normally used in air rather than in vacuum and depends on polymethylmethacrylate supports for thermal insulation. It is also equipped with a calibration heater. The front surface of the absorber is corrugated, with concentric grooves just sufficient to restrict electron backscatter from the aluminum to match approximately the backscatter from a totally absorbing stack of plastic, radiochromic-dye films. With appropriate corrections for radiant heat losses, the system is capable of measuring collimated electron beam power absorbed by the calorimetric body, even for very short high-intensity single pulses, such as those accelerated from a field-emission generator.

The second phase of this program consists of the first basic chemical studies of the response of liquid solutions of organic triphenylmethane dye precursors, which up to now have been used successfully for measuring gamma-ray doses in the range from  $10^3$  to  $10^5$  rads. Since there is no satisfactory chemical system available for measuring doses between  $10^5$  and  $10^6$  rads, attempts were made to extend the response of the triphenylmethane dye systems by at least one order of magnitude. A detailed study is being carried out on the system consisting of pararosanine cyanide (1 to 8mM), in 2-methoxy ethanol or aqueous acetic acid as activating solvents for the radiochromic dye. The investigations have shown so far that the radiation yield for the dye formation varied markedly with the acidity and with the concentrations of oxygen and the mild oxidizing agent. The linear response of the liquid radiochromic dye systems could be significantly extended by the addition of a proper mild oxidizing agent. In 2-methoxy ethanol solutions, saturated with pure oxygen, the dye formation is linear with absorbed dose to about  $5 \times 10^5$  rads, while the addition of 30 mM of nitrobenzene extends the useful range up to  $10^6$  rads. Extending the linearity of the dye formation caused a slight reduction in sensitivity. In aqueous acetic acid solutions the effect of oxygen and nitrobenzene concentration on the dye formation was similar: by increasing the oxygen and nitrobenzene concentration, the linear response of the dye formation was extended, while the G-value decreased. Details of this work were given in a paper presented at the recent International Atomic Energy Agency Symposium (Vienna, April 1972).

Preliminary experiments were made with these systems in pulsed electron beams in order to study the response to very short pulses and to learn more about the basic chemical kinetics of the dye formation.

List of Publications that Resulted from the Project

1. B. Radak, P. E. Hjortenber<sup>g</sup> and N.W. Holm, "A Calorimeter for Absolute Calibration of Thin Film Dosimeters in Electron Beams," Paper No. SM-160/31, IAEA Symposium of Dosimetric Techniques in Agriculture, Industry, Biology, and Medicine, Vienna (April 1972), (in press).
2. I. Draganic<sup>ć</sup>, "Some Current Tendencies in Chemical Dosimetry," Paper No. SM-160/93, ibid (in press).
3. W. L. McLaughlin, M. Kosanic<sup>ć</sup> and I. Draganic<sup>ć</sup>, "Extending the Linear Gamma-ray Response of Pararosaniline Cyanide Solutions from 0.1 to 1 Megarad," Paper No. SM-160/93a, ibid (in press).



## APPENDIX I

### TWO EXAMPLES OF ATTRACTIVE OPPORTUNITIES FOR NEW COOPERATIVE NBS RESEARCH GRANTS

#### Example 1

Title: PHASE DIAGRAMS FOR CERAMISTS: Investigations of the Systems  
 $\text{BaO-CeO}_2\text{-TiO}_2$  and  $\text{BaO-Al}_2\text{O}_3\text{-TiO}_2$

Principal Investigator: Dr. Drago Kolar

Institution: Jožef Stefan Institute, Ljubljana

NBS Monitor: Dr. E. M. Levin

NBS Institute or Center and Division: Institute for Materials Research  
Inorganic Materials Division

#### Summary of Description of Work Proposed

In the past ten years, materials science and technology have grown rapidly. One of the most important sources of information regarding a materials system is its phase diagram. Therefore, there is a great need for accurate phase diagrams for different systems. In spite of extensive research in this field, the knowledge of phase equilibria in several important systems is still lacking. This is even true for such extensively investigated systems such as  $\text{BaO-TiO}_2\text{-M}_x\text{O}_y$ .

Barium titanate is a well known ferroelectric material. The ferroelectric and piezoelectric properties of barium titanate have been widely studied for capacitor and transducer applications. The level, as well as the temperature and electric field dependence, of the relative permittivity of  $\text{BaTiO}_3$  is influenced by structure modifications through solid solutions and by some physical properties such as density and grain size.  $\text{BaTiO}_3$  generally does not sinter readily; however, the sinterability may be improved by the addition of some elements. Other additives may be used as grain growth inhibitors.

Many references describing the beneficial or deleterious influence of different additives on the electrical properties of  $\text{BaTiO}_3$  may be found in the literature. However, a great deal of work turns out to be of an empirical nature and, in most cases, it has not been determined if there is a true solid solution, a multi-phase equilibrium system, or an incomplete reaction. Hence it is difficult to correlate the properties of these materials with their chemical composition and physical state.



It is proposed to carry out the investigations using the systems  $\text{BaO-TiO}_2\text{-Al}_2\text{O}_3$  and  $\text{BaO-TiO}_2\text{-CeO}_2$ . To start with, binary diagrams  $\text{BaTiO}_3\text{-BaAl}_2\text{O}_4$  and  $\text{BaO-CeO}_2$  will be constructed. To accomplish this, standard dynamic and static methods will be used, such as room temperature and high temperature X-ray techniques, microprobe analysis, metallography, differential thermal analysis, dilatometry, etc.

The successful accomplishment of the first task will enable the next stage of the investigation to proceed, namely, the construction of other tie-lines in the mentioned ternary systems from the new data and the data already in the literature for  $\text{BaO-TiO}_2$ ,  $\text{BaO-Al}_2\text{O}_3$ , and  $\text{Al}_2\text{O}_3\text{-TiO}_2$ .

The results obtained will contribute to the knowledge of phase relationships in the systems involved and will therefore be of general interest to inorganic-materials scientists and ceramists. The results obtained will be of practical value, especially to those interested in preparation and properties of ceramics for electronic applications.

Results and Implications to Date  
(Based on work supported entirely by the  
Jozef Stefan Institute)

Previous research indicated that by combining  $\text{BaTiO}_3$ ,  $\text{CeO}_2$  and  $\text{Al}_2\text{O}_3$ , materials with interesting electronic properties may be prepared. It is known that the incorporation of  $\text{CeO}_2$  into  $\text{BaTiO}_3$  improves the electric properties of  $\text{BaTiO}_3$  mainly by preventing the grain growth. On the other hand, it is claimed that  $\text{Al}_2\text{O}_3$  has a deleterious effect on  $\text{BaTiO}_3$  ceramics. Preliminary investigations of the  $\text{BaTiO}_3\text{-Al}_2\text{O}_3$  system indicated the formation of  $\text{BaAl}_2\text{O}_4$  and  $\text{BaTi}_4\text{O}_9$ . In more detailed research the tie-lines in the system  $\text{BaO-Al}_2\text{O}_3\text{-TiO}_2$  were to be investigated, as for example, in the binary diagram  $\text{BaTiO}_3\text{-BaAl}_2\text{O}_4$ . Data on the binary systems along the tie-lines in the ternary system  $\text{BaO-TiO}_2\text{-CeO}_2$  are also lacking. As a first step, the binary diagram  $\text{BaO-CeO}_2$  will be investigated.

In the next stage other possible tie-lines in both systems,  $\text{BaO-CeO}_2\text{-TiO}_2$  and  $\text{BaO-Al}_2\text{O}_3\text{-TiO}_2$ , will also be investigated so as to enable, with the help of available data, the construction of the ternary phase diagrams.

In the course of the proposed research, standard dynamics and static methods will be used, such as differential thermal analysis, dilatometry, room temperature and high temperature X-ray techniques, microprobe analysis, metallography, etc.

However, the understanding of the reactions involved calls for more detailed knowledge of equilibrium phase diagrams.

Recent results of the ceramic laboratory of the Jožef Stefan Institute in the field of phase equilibria determinations are briefly described below:

Sub-solidus equilibria involved in the ternary systems,  $\text{BaO-TiO}_2\text{-CeO}_2$ ,  $\text{BaO-TiO}_2\text{-Al}_2\text{O}_3$ , and  $\text{BaO-TiO}_2\text{-GeO}_2$  have been investigated. Various tie-lines existing in these systems were established. Three new ternary compounds have been identified in the system  $\text{BaO-TiO}_2\text{-Al}_2\text{O}_3$ . Work on the crystal structures and melting behavior of these compounds is in progress.

Several binary systems, namely  $\text{BaO-CeO}_2$ ,  $\text{TiO}_2\text{-CeO}_2$ ,  $\text{BaTiO}_3\text{-CeO}_2$ ,  $\text{BaTiO}_3\text{-BaCeO}_3$  and  $\text{BaTiO}_3\text{-BaGeO}_3$  have been studied, and the solid-liquid equilibria involved were established.

Further work indicated that, in all the ternary systems mentioned above, liquid phases, probably ternary eutectics, occur near the  $\text{BaTiO}_3$  composition. It is expected that the formation of liquid at considerably lower temperatures would have a strong influence on the ceramic (e.g. density and microstructure) as well as electrical properties of  $\text{BaTiO}_3$ . Further work is now in progress to develop a clear understanding of the phase equilibria involved in these ternary systems.

From a practical point of view, the results obtained so far have enabled us to prepare  $\text{BaTiO}_3$  ceramic capacitors having a C of over  $200,000 \text{ pF/cm}^2$ . Another important aspect of this project deals with the collection of phase diagram data, particularly those data reported by Eastern European scientists. Such data are largely missing from the latest NBS publication of the 1969 edition of "Phase Diagrams for Ceramists," and would significantly increase the value and utility of this volume. About 25 percent of the effort will be devoted to identifying and reviewing publications in selected Eastern European journals and Symposium Proceedings related to phase equilibria work. Much of this work especially publications arising from Symposium presentations are undetected

and unreported in the NBS and other publications produced outside of Eastern Europe.

Publications relating to the investigation on the systems BaO-TiO<sub>2</sub>-CeO<sub>2</sub>,  
BaO-TiO<sub>2</sub>-GeO<sub>2</sub> and BaO-TiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> covering the period 1970-1972

1. M. Buh, D. Kolar and J.P. Guha, "The Effect of GeO<sub>2</sub> on the Microstructure, Density and Dielectric Properties of BaTiO<sub>3</sub>". Presented at the 2nd Yugoslavian Conference on Electronic Materials, held at Ljubljana, Yugoslavia in October 1970.
2. M. Buh, D. Kolar and J.P. Guha, "The Influence of Al<sub>2</sub>O<sub>3</sub> on the Microstructure, Densification and Dielectric Properties of BaTiO<sub>3</sub>". Proceedings of the 4th Conference on Electronic Ceramics, held at Spindleruv Mlyn, Czechoslovakia in May 1971.
3. J. P. Guha, D. Kolar and V. Urbanc "DTA and X-ray Analyses on Phase Transitions and Compatibility Relationships in the Pseudobinary System BaSiO<sub>3</sub>-BaGeO<sub>3</sub>." Proceedings of the 3rd International Conference on Thermal Analysis, held at Davos, Switzerland in August 1971.
4. J. P. Guha and D. Kolar "Phase Equilibria in the System BaO-CeO<sub>2</sub>." Journal of Materials Science, 6, 1174-1177 (1971).
5. J. P. Guha and D. Kolar "Identification of BaO.4TiO<sub>2</sub>.Al<sub>2</sub>O<sub>3</sub> in the System BaO-TiO<sub>2</sub>-CeO<sub>2</sub>." Journal of the American Ceramic Society, 55, 55 (1972).
6. J. P. Guha and D. Kolar "Phase Equilibria in the System BaTiO<sub>3</sub>-BaGeO<sub>3</sub>." Journal of Materials Science, 7, 1192-1196 (1972).
7. J. P. Guha and D. Kolar "Subsolidus Equilibria in the System BaO-TiO<sub>2</sub>-CeO<sub>2</sub>." Journal of the American Ceramic Society (Scheduled for publication in the January 1973 issue).
8. D. Kolar, J. P. Guha and M. Buh, "Ceramic and Dielectric Properties of Selected Compositions in the BaO-TiO<sub>2</sub>-CeO<sub>2</sub> system." Selected for presentation in the meeting on Electrical Magnetic and Optical Ceramics organized by the British Ceramic Society, to be held in London in December 1972.



## Example 2

Title: ELECTROCHEMICAL BEHAVIOR OF SOME BLOOD PROTEINS IN RESPECT TO THROMBOSIS  
AND BLOOD COAGULATION MECHANISMS

Principal Investigator: Dr. Ljerka Duić

Institution: Institute for Chemical Technology, University of Zagreb

NBS Monitor: Dr. Robert R. Stromberg

NBS Institute or Center and Division: Institute for Materials Research  
Polymers Division

### Summary Description of Project Goals

A better understanding of the interactions between synthetic implant materials, which are placed in the body to replace or assist diseased or damaged organs and tissues, and the components in blood serum is essential if improved materials are to be made available to affected people. Because key information on such interactions are incomplete or lacking, physicians have been unable to precisely specify the properties of the materials which they require for certain surgical applications. Also, manufacturers of polymeric, metallurgical and inorganic materials and products for use as synthetic implant have largely been unable to obtain information and specifications from which to prepare and manufacture non-thrombogenic, long lasting implants. Indeed, one of the pressing problems in this field is that of a high tendency of certain synthetic materials to cause blood clotting when used in the human body because of electrochemical phenomena. Thus, one of the crucial areas requiring attention is work on the electrochemical steps involved in thrombosis.

This proposal presents a well developed plan for investigating the electrochemistry of selected blood proteins at the electrode-solution interface with the promise of obtaining accurate data on electrosorption and charge transfer processes. By obtaining quantitative information on which proteins in blood serum are involved in electrosorption and charge transfer processes and which are not, and coupling this with physical and chemical information on materials used as implants, one can derive a fuller understanding of the interactions involved. This in turn should make it possible to develop meaningful standards and specifications for synthetic implants with regard to these important parameters.

Although this proposal has not yet received support from the Special Foreign Currency Program, a significant start has been made already on this problem using entirely Yugoslav funds. It is now clear that the electrochemical behavior of each blood coagulation factor needs to be determined separately if the extent of electrochemical reactions in thrombosis are to be fully explained. Cyclic-voltammetry studies by the principal investigator and co-workers have already shown that some single blood coagulation factors such as prothrombin, thrombin and fibrinogen (see list of publications) participate in electrochemical reactions. It is proposed to conduct a detailed study of the electrochemical behavior of four proteins: prothrombin, thrombin, fibrinogen and albumin. Such studies would add important information about these proteins and their reactions and would directly benefit the project at the National Bureau of Standards which is investigating these systems using an entirely different technique, namely, ellipsometry. There is a strong indication that data obtained by both techniques, at least, will be necessary to resolve the problem of thrombosis induced by synthetic implants.

List of Publications that Resulted from this Project

1. L. Duic, S. Srinivasan and P. N. Sawyer, "Electrochemical Behavior of Blood Coagulation Factors: Prothrombin and Thrombin" (in press).
2. N. Ramasamy, M. Ranganathan, L. Duic, S. Srinivasan and P.N. Sawyer, "Electroactivity of Fibrinogen: Ellipsometric and Potentiodynamic Studies" (in press).



## APPENDIX II

### NBS PROGRAM STRUCTURE

#### A. Promoting Accurate, Meaningful and Compatible Scientific and Technical Measurements

1. Physical measurement methods, units and standards
  - a. Length and wavelength
  - b. Dimensional metrology (including surface topography)
  - c. Mass, volume, density
  - d. Force, hardness
  - e. Acceleration, vibration, shock
  - f. Acoustic quantities
  - g. Flow characterization (liquid and gas)
  - h. Humidity
  - i. Pressure
  - j. Temperature
  - k. Radiometry, photometry, spectrophotometry
  - l. Far UV radiometry
  - m. X-ray,  $\gamma$ -rays, electron beam and dose measurements
  - n. Radioactivity measurements
  - o. Neutron field and dose measurements
  - p. Electrical (dc - lf)
  - q. Electromagnetic (rf, microwave and millimeter wave)
  - r. Laser quantities
  - s. Plasma quantities
  - t. Optics and imaging (light and electron)
  - u. Opto-electronics
  - v. Cryoelectronic techniques
  - w. Frequency and time
2. Measurement transfer services
  - a. Tests and calibrations - development
  - b. Tests and calibrations - operation
  - c. Measurement assurance - development
  - d. Measurement assurance - operation
  - e. Measurement instrumentation services
  - f. Evaluation of the national measurement system
3. Time and frequency dissemination services
  - a. Exploration and development
  - b. Broadcast services
4. (Not used)
5. Standard reference data
  - a. Data evaluation
  - b. Coordination of National Standard Reference Data System
  - c. Publication and distribution
  - d. Data systems design
  - e. Information services

6. Standard reference materials
  - a. Production and certification
  - b. Operation
7. (Not used)
8. Materials measurement methods and standards
  - a. Materials preparation
  - b. Materials characterization
  - c. Feasibility of new reference materials
  - d. Properties of materials
9. Properties of matter
  - a. Measurement system data - physical constants
  - b. Measurement system data - benchmark data
  - c. Essential technical data
10. Exploratory research

B. Promoting More Effective Use of Science and Technology

1. Materials science and technology
  - a. Metallurgical materials
  - b. Polymeric materials
  - c. Inorganic materials
  - d. Composite materials
  - e. Dental materials
  - f. Chemical materials
2. Environmental pollution measurement methods and standards
  - a. Air
  - b. Water
  - c. Solid waste
  - d. Noise
  - e. Electromagnetic
3. Building Technology
  - a. Building codes and standards participation
  - b. Professional and industrial information exchange
  - c. Consulting services
  - d. Physical and functional environment of buildings
  - e. Strength and safety in structures
  - f. Durability of building materials
  - g. Building systems
  - h. Housing technology
4. Computer science and technology
  - a. Utilization
  - b. Teleprocessing
  - c. Services
  - d. Specific applications

4. (computer science and technology cont.)

- e. Exploratory development
- f. Automation technology
- g. Technical and information services

5. Analysis of technical systems

- a. Development of new methods
- b. Applications

6. Electronic technology

- a. Development of measurement methods
- b. Instrumentation applications
- c. Information services

7. Cryogenic technology

8. Technology utilization analysis

- a. Innovation policy and encouragement
- b. Inventor assistance
- c. Policy studies
- d. Innovation education (inactive)

9. Failure avoidance

- a. Materials
- b. Structures and machinery
- c. Disaster analysis
- d. Failure analysis and technology transfer

10. (Not used)

11. Nuclear and radiation technology

12. Illumination technology

C. Promoting Strength in the Economy and Equity for Buyer and Seller in Trade

1. Voluntary engineering standards

- a. Standards policy and coordination
- b. International standards committee participation
- c. Domestic standards committee participation
- d. Department of Commerce voluntary standards
- e. Standards information service

2. Measures of quantities important to commerce

- a. Weights and measures
- b. Practical industrial measurements

3. Product performance

- a. Research and performance test development
- b. Test services
- c. Standards for government

4. Testing laboratory evaluation
5. Fair packaging and labeling

D. Standards and Test Methods for Protection of the Public from Specified Hazards

1. Fire research
  - a. Flammable fabrics
  - b. Fire research and safety
2. Radiation safety
  - a. Radioactivity
  - b. X-rays and other ionizing radiation
  - c. Microwave radiation
  - d. Laser radiation
  - e. Ultrasonic radiation
3. Product safety
  - a. Standards support and participation
  - b. Hazard analysis
  - c. Research and test method development
  - d. Information services

E. Technical Information Services

1. Information evaluation, documentation, and reference services
  - a. Technical information analysis centers
  - b. Documentation, reference and information services
2. Symposia, technical meetings, and training courses
3. Research and development in information sciences
4. Consulting and advisory services

F. Central Technical Support

1. Operation of major facilities
  - a. Linear Accelerator
  - b. Nuclear reactor
  - c. Other
2. Facilities modifications and improvements
3. Mathematical supporting services
4. Engineering and instrumentation support
5. Supporting services for other agencies

G. Experimental Technology Incentives Program

## SFCP (PL 480) BACKGROUND HISTORY

Since the Fiscal Year (FY) 1961 the National Bureau of Standards has had an active special foreign currency program (SFCP) in India, Israel and Pakistan. During 1970, Yugoslavia was added to the list of countries and Poland and the Arab Republic of Egypt in 1972.

The relevant US laws, such as PL-480 of the 83rd Congress pertaining to the use of SFCP funds, challenge imaginative implementation at the same time as they offer the opportunity for international collaboration to universal benefit. In the mission area of NBS and with the available countries, Dr. Lewis M. Branscomb, former Director of NBS, had called for an effective program. To this end, Mr. H. Steffen Peiser, as program manager, under the supervision of Dr. E. L. Brady, Associate Director for Information Programs, has received wide-ranging support, which is hereby gratefully acknowledged.

Stated Policy for NBS/SFCP Grants

NBS/SFCP grants provide supplementary resources that provide an appropriate means for accomplishing NBS objectives, consistent with U.S. foreign policy objectives. NBS criteria for this purpose are:

- "1. All projects must be of scientific or technological excellence.
2. All projects must be within the mainstream of NBS interests or contribute directly to NBS effectiveness..
3. All projects must benefit both the U.S. and the participating countries:
  - (a) NBS/SFCP funds must not merely replace otherwise available country funds; and
  - (b) Individuals or institutions abroad must not become primarily dependent upon NBS for long term support.
4. All projects must be followed closely by NBS staff members working in related areas. Thus, one or more NBS staff should maintain a continuing direct relationship with the foreign person or group. This relationship should be strengthened by visits in either or both directions.



5. NBS involvement with a technical institution of another nation should be consistent with U.S. foreign policy. For example, relationships with sister institutions, staff exchanges, and visits should be carried on in the context of overall U.S. objectives in its relationships with the other country.
6. NBS staff travel to participating foreign countries should be exploited, when practical, by suitable additional itinerary to identify additional targets for bilateral benefit or to provide additional strength to existing relationships. Such travel should be coordinated with the NBS/SFCP Manager."

#### Bureau Level Management

Mr. H. Steffen Peiser has been designated the NBS/SFCP Manager by the Director.

As such he has the following responsibilities and authorities:

- "1. Makes final decisions on grant proposals, allocates funds, and signs SFCP grant documents.
2. Serves as the principal point of contact with:
  - a. The Director on NBS/SFCP policy,
  - b. The Institutes and Centers on specific proposals and program planning,
  - c. The Budget Division on all budget related matters,
  - d. The Procurement Section on grant execution and administrative matters,
  - e. The Office of International Relations on matters of channelling communications and relations with the Department of State.
3. Authorizes all foreign travel related to the activated or potential NBS/SFCP grants."

#### Institute Level Management

Each major organizational unit has the following responsibilities:

- "1. Technically evaluates proposals and determines whether the proposals meet Criteria 1, 2 and 4 of the SFCP policy statement for support under the program.
2. Recommends proposals to the SFCP Manager for support and makes priorities known to him.
3. Assures that there is continuing involvement by the designated monitor once a grant has been let.
4. Advises and assists the SFCP Manager in formulating program plans and budget requests."

# APPENDIX IV

## LIST OF SFOP GRANTS IN YUGOSLAVIA

Date First Authorized	Title	Principal Investigator	NBS Office, Center or Institute	NBS Monitor	Funds Committed Prior to Dec.1972 (\$K)	Page(s)
9/15/70	Determination of Impurity Levels in Analytical Grade and Ultrapure Chemicals by Activation Analysis Using Triga Reactor	Lado Kosta	IMR	W.W. Meinke	70.0	7
10/1/70	Bulk Composition and Surface Properties of Glass	Velimer Pravdic	IMR	W. Haller	66.5	33
4/25/71	Pattern Recognition & Processing	Vladimir Bonacic	CCST	T.N. Pyke	12.5	41
8/1/71	Radiation Dosimetry and Radiation Chemistry	Ivan Draganic	IBS	W.M. McLaughlin	40.0	45
8/9/71	Physics of Ionized Gases	Nikolla Konjevic	IBS	W.L. Wiese	20.0	21
9/28/71	Electrochemical Study of Metal Ion-ligand Interaction in Non-aqueous Solutions by Investigation of Electrochemical Property of ligand	Ivan Piljac	IMR	J. K. Taylor	20.0	15
6/30/71	Syntheses and Properties of Fluoride Complexes	J. Slivnik	IMR	T. D. Coyle	20.0	36
11/12/71	Development and Application of Ultrasensitive Magnetic Resonance	R. Blinc	IMR	D. H. Reneker	20.0	11
4/18/72	A Modular Computer System for Real-time Data Reduction and Control in Biomedical Experiments	Z. Damjanovic	CCST	T. N. Pyke	20.0	43
4/1/72	Mass Spectrometric Studies of High Temperature Equilibria	K. F. Zmbov	IMR	J. W. Hastie	20.0	17

Date First Authorized	Title	Principal Investigator	NBS Office, Center or Institute		Funds Committed Prior to Dec. 1972 (k\$)		Page(s)
				NBS Monitor			
4/25/72	Part I: Electron-atom Excitation Cross-Section Measurements	V. Urosevic	IBS	G.H.Dunn (Boulder)	20.0	29	
	Part II: Electron-atom Elastic Scattering	M. Kurepa	IBS	C. Kuyatt		25	
4/28/72	Measurement of f-values in Ultraviolet and Visible Region and Study of Continuous Emission of Hydrogen Near the Balmer Limit	D.Muller V. Vujnovic	IBS	W. L. Wiese	20.0	27	
6/1/72	International Summer School on Radionuclides Metrology	Dj. N. Bek-Uzarov	IBS	W. B. Mann	4.29	5	

## APPENDIX V

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University of Zagreb .....	15, 27

## APPENDIX VI

## ALPHABETIC LIST OF PRINCIPAL INVESTIGATORS

Principal Investigator	University, Institute or Laboratory	Page(s)
Bek-Uzarov, Dj. N.	Boris Kidrič	5
Blinć, R.	Jožef Stefan	11
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Kurepa, M.	University of Belgrade (Institute of Physics)	25
Müller, D.	University of Zagreb (Institute of Physics)	27
Piljac, I.	University of Zagreb	15
Pravdić, V.	Rüdjer Bošković	33
Slivnik, J.	Jožef Stefan	36
Urosević, V.	University of Belgrade (Institute of Physics)	29
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Zmbov, K. F.	Boris Kidrič	17



## APPENDIX VII

## ALPHABETIC LIST OF NBS MONITORS

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Dunn, G. H.	274.00 (Boulder)	29
Haller, W.	313.02	33
Hastie, J. W.	313.01	17
Kuyatt, C. E.	232.05	25
Mann, W. B.	242.00	5
McLaughlin, W. M.	243.02	45
Meinke, W. W.	310.00	7
Pyke, T. N.	650.02	41, 43
Reneker, D. H.	311.03	11
Taylor, J. K.	310.00	15
Wiese, W. L.	232.07	21, 27

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7. AUTHOR(S) H. Steffen Peiser, Samuel E. Chappell, Emanuel Horowitz, Harvey Yakowitz and Doris Bluebond		8. Performing Organization	
9. PERFORMING ORGANIZATION NAME AND ADDRESS  NATIONAL BUREAU OF STANDARDS DEPARTMENT OF COMMERCE WASHINGTON, D.C. 20234		10. Project/Task/Work Unit No.	
		11. Contract/Grant No.	
12. Sponsoring Organization Name and Address  Same as No. 9		13. Type of Report & Period Covered Final	
		14. Sponsoring Agency Code	
15. SUPPLEMENTARY NOTES			
16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)  An overview is given of grants awarded by the National Bureau of Standards under the Special Foreign Currency Program (SFCP) in Yugoslavia, authorized by Public Law 480 and other legislation. Each grant is identified by title, principal investigator, institution in Yugoslavia, NBS monitor, and the monitor's organizational unit within NBS. The work is then described briefly under the three headings "Summary Description of Project Goals," "Results and Implications to Date" and "List of Publications that Resulted from the Project." To demonstrate the relevance of such grants to the programs of NBS, the grant descriptions are ordered by the elements of that Program Structure. The significance and purpose of the NBS/SFCP grant program are discussed in the Foreword, the Introduction and an Appendix. The NBS monitors and the program manager judge this grant program to have had a high benefit to cost ratio from the viewpoint of NBS.			
17. KEY WORDS (Alphabetical order, separated by semicolons) Binational research cooperation; international scientific cooperation; physical science research administration; research planning; scientific research abstracts; Special Foreign Currency Program; Yugoslavia science and technology.			
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